

NATIONAL BUREAU OF STANDARDS

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U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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U.S. DEPARTMENT OF COMMERCE

C. R. Smith, Secretary

NATIONAL BUREAU OF STANDARDS

A. V. Astin, Director

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**Cover:** In determining the refractive index of hydrogen, D. E. Diller photographs interference patterns produced by passing monochromatic light through liquid hydrogen. (See page 207.)

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# SPALLING OF PORCELAIN-ENAMELED ALUMINUM

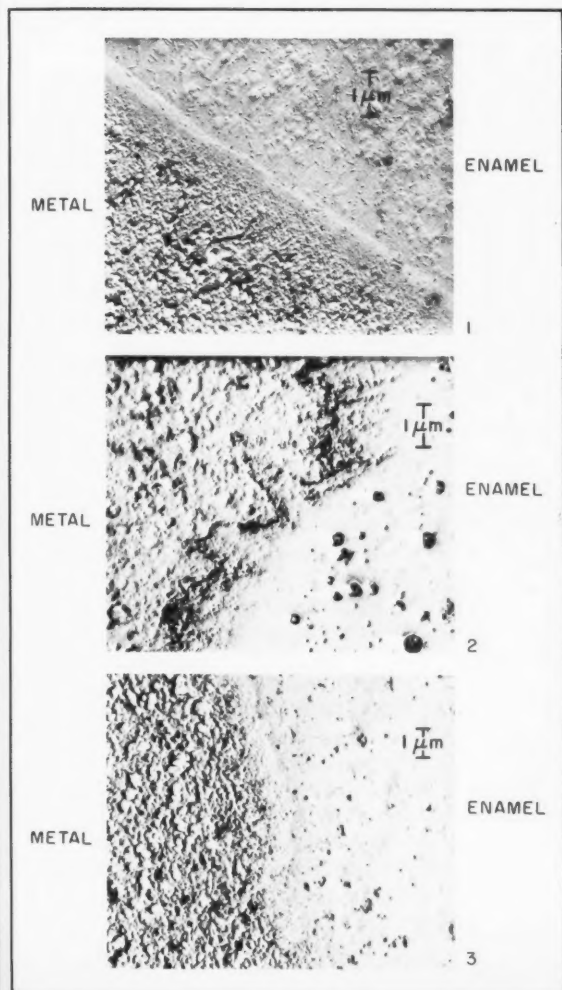
The results of a recent NBS study indicate that spalling in porcelain-enameled aluminum is probably caused by the corrosive attack of moisture and moisture-salt mixtures at the enamel-metal interface. The nature and cause of spalling (the chipping or flaking of porcelain enamel from its metal substrate) is being investigated at the NBS Institute for Applied Technology. Albert L. Gugeler, Porcelain Enamel Institute Research Associate at NBS from the Ferro Corporation, is conducting this investigation into the mechanisms of porcelain enamel adherence to aluminum. The purpose of the study is to develop not only a means of preventing spalling but also a more accurate test for predicting spall resistance of various aluminum alloys.

Since the end of World War II, increasing attention has been given to the porcelain enameling of aluminum, particularly in the fields of architecture and cookware. This is because pure aluminum is relatively easy to enamel and such enameling provides a permanent, weatherproof, and abrasion-resistant color on the aluminum.

Since commercially pure aluminum is fairly soft, it is alloyed with other elements to increase its strength and enhance its other properties. But the resulting alloys are more susceptible to spalling than the pure metal, and this has become one of the major problems of the porcelain-enameled aluminum industry. To prevent spalling, various pre-enameling treatments have been used with the higher-strength alloys and have proven beneficial. These pre-enameling cycles normally include a chromating treatment, which makes it possible to enamel several higher-strength aluminum alloys with confidence. Many otherwise desirable aluminum alloys, however, cannot be enameled by any known means.

Several tests (including ASTM C-486) have been developed to predict the resistance of a particular porcelain-enameled aluminum system to spalling. The spall tests consist of immersion of enameled specimens in

*continued*



*Commercially pure aluminum cleaned by firing before enameling (lower left) appears to make direct contact with the porcelain enamel (upper right).*

*An aluminum alloy with 3.5 percent magnesium, prefired before enameling, shows an intermediate zone between the metal and enamel; this sample spalled almost completely.*

*An aluminum alloy with 1 percent magnesium indicates an intermediate zone after enameling; this sample spalled under test. However, with proper pre-enameling treatment and chromating, this aluminum alloy does not spall.*

## SPALLING *continued*

chloride solutions for specified periods of time. ASTM C-486 utilizes a 5 percent solution of ammonium chloride at room temperature, with an immersion time of 96 hours. Another test sometimes used consists of a 1 percent solution of antimony trichloride at room temperature, with an immersion time of 20 hours. Although these tests provide excellent guidelines, they do not always predict spall resistance with 100 percent accuracy. For this reason, the Porcelain Enameled Aluminum Council of the Porcelain Enamel Institute is sponsoring a Research Associate program at NBS. The Aluminum Council feels that if the mechanism of adherence and the nature and cause of spalling can be determined, then a more accurate test for spall resistance can be developed.

In the present study, a standard porcelain enamel composition has been used on a variety of aluminum alloy samples. The alloy tested and the pre-enameled methods have been varied with some interesting results. The commercially pure alloys can be enameled without subsequent spalling,

simply by cleaning the metal before enameling. The cleaning may be accomplished either by chemical action which removes contaminants such as greases, oils, and dirt; or by firing the samples at 538 °C (1000 °F) for approximately 10 minutes which burns off the surface contaminants.

Alloys containing magnesium, however, present some problems. None can be enameled with 100 percent success after simple cleaning, and some cannot be enameled even with more extensive pre-enameled and chromating procedures.

Electron microprobe examinations were made on a number of the magnesium-bearing alloys which had been porcelain enameled without prior chromating pretreatments, and which showed spalling. In a number of these alloys, there was a build up of magnesium or a compound of magnesium at the interface between the porcelain enamel and the aluminum. It was also found that if magnesium metal was vapor deposited in thin films on the surface of commercially pure aluminum alloys, these alloys would spall after enameling, unless the magnesium film was covered with a vapor-deposited chromium film.

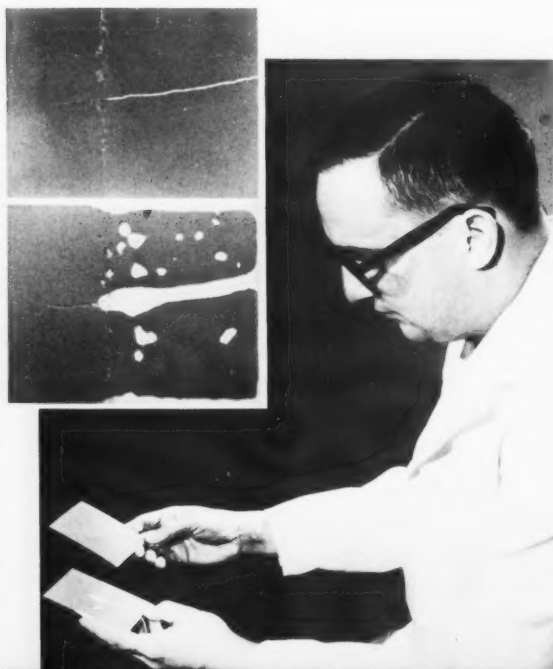
Another series of enameled samples was examined in cross section with the electron microscope. As the samples represented a range of alloys and pre-enameled treatments, this series covered a range of spall characteristics. Those that exhibited no spalling after enameling and testing showed an interface where the enamel and metal were in intimate contact. This was also true of commercially pure alloys that were cleaned by firing before enameling.

Quite different results were obtained, however, with alloys containing magnesium. One containing 3.5 percent magnesium spalled almost completely when subjected to spall tests after firing, even when chromated. An alloy containing 1 percent magnesium also exhibited spalling, although not to the same extent as the 3.5 percent-magnesium alloy. In both magnesium-bearing alloys, where spalling occurred there was no smooth transition or direct contact between the enamel and the metal, but rather a band or zone of intermediate structure. On the other hand, when the 1 percent-magnesium alloy had proper pre-enameled treatment and was chromated, no spalling occurred. Electron micrographs of cross sections of this type of system were taken by David Ballard of NBS and show an interface similar to that of the enameled pure aluminum alloy.

Early evidence seems to indicate that the zone of intermediate structure between the enamel and the metal is highly susceptible to corrosive attack by moisture and moisture-salt mixtures. Corrosion of this type, occurring initially along exposed edges or at minute discontinuities in the porcelain enamel, might result in enough pressure to lift or force the enamel away from the metal.

This research is continuing with special emphasis on the intermediate zone. In particular, the investigation is aimed at determining what constitutes this zone, why the zone is present in some systems and not others, and how magnesium affects the zone formation.

*Albert Gugeler examines two porcelain-enameled aluminum specimens that were tested for spalling by scoring and then immersing in a spall test solution. The aluminum specimen (top) did not spall at the scoring; however, the aluminum-alloy specimen, which was improperly pretreated, spalled at the scoring and elsewhere.*





# METAL BEHAVIOR STUDIED AT LOW TEMPERATURES

The rapid advances of cryogenic technology in aerospace applications, electronics, and high-energy physics have necessitated a basic understanding of the physical nature of materials suitable for use at low temperatures. Of particular interest are those metallic alloys that remain both strong and ductile at low temperatures and thus are well suited for cryogenic equipment.

As part of a comprehensive materials research program at the National Bureau of Standards, R. P. Reed of the NBS Cryogenics Laboratory (Boulder, Colo.) has been investigating the crystallographic changes that occur in some metallic alloys when they are cooled to extremely low temperatures or subjected to stresses at low temperature so that they become plastically deformed.<sup>1</sup>

Although some metals, notably the ordinary carbon (structural) steels, become brittle and tend to break when stressed at very low temperatures, others, including the austenitic stainless steels and some iron-nickel alloys, do not exhibit this "brittle fracture" under cryogenic conditions. Instead, they are ductile, deforming relatively easily to relieve applied stresses.

In some metals, during either cooling or plastic deformation, a partial transformation occurs in the crystal structure to a form termed "martensite." This new crystallographic structure is produced by the coordinated shearing, through small distances, of millions of atoms into new positions. The martensite crystals form in even arrays, on definite planes, because the atoms are more stable in the new positions than in the old. It is this change in crystal structure that is responsible for the high work-hardening capacity of many low-temperature alloys. Although the martensite transformation, which is the sole change in crystallographic structure that can occur in the solid state at low temperatures, has been observed since the last part of the 19th century, no complete theory for the transformation has as yet been formulated.

The long-range objective of the study is an understanding of the fundamental characteristics of martensite. Optical-microscopy, x-ray diffraction, and electron-microscopy techniques have been used to study the crystallography and kinetics of the martensite transformation. The crystallographic studies have shown growth patterns,



*R. P. Reed adjusts the electron microscope during a study of metal behavior at cryogenic temperatures.*

lattice parameters, and final orientations of the martensite crystals. Kinetic studies have revealed information on the conditions under which the transformations form and include the parameters of temperature, time, applied stress, and chemical composition.

The first phase of this continuing program has been concerned with investigation and documentation of the crystallography and kinetics of the austenitic stainless steels,<sup>2</sup> and determination of their magnetic and mechanical properties as influenced by the martensite transformation.

The second phase of the study is concerned with the transformation characteristics of a series of iron-nickel alloys. By varying the percentage of nickel in the alloy specimens, it has been possible to study the correlation of martensite crystallography and morphology as a function of chemical composition. To date, the martensite transformation has been documented in the chemical range from 24 to 36 percent Ni by weight. For the first time, results showed (1) that martensite morphology, the midrib plane, and internal twinning of iron-nickel alloys are critically dependent on chemical composition; and (2) that the growth, or plate formation, is dependent on the local temperature.

<sup>1</sup> Plate-like martensite in Fe-Ni alloys, by R. P. Reed, *Acta Met.* 15, 1287 (1967); see also, Martensitic transformation products in Fe, 26 to 30 wt% Ni alloys, by R. P. Reed, *Trans. AIME* (to be published).

<sup>2</sup> Stress-induced martensitic transformations in 18Cr-8Ni steel, by R. P. Reed and C. J. Guntner, *Trans. AIME* 230, 1713 (1964).

*L. D. Driver adjusts the internal reference voltage of the radiofrequency signal stabilizer he developed. It makes it possible to obtain a highly stable rf signal without using elaborate equipment or improvised circuitry.*



## STABILIZING RADIOFREQUENCY POWER

### *Simple Device Improves Signal Quality*

In many calibration and other laboratories it is often necessary to stabilize the output level of radiofrequency generators. To achieve a high degree of rf signal amplitude stability, it is common practice to use either a special, high-cost, stabilized rf generator or a "benchful" of auxiliary electronic gear in combination with an ordinary rf generator. In either case, signal stability is achieved at the expense of time and money. Recently, however, a device has been developed which is small in size and economical in cost and is capable of automatically stabilizing the output signals of ordinary rf generators. This device is the work of L. D. Driver of NBS.

Although other methods for stabilizing generator-signal amplitude have been available for some time, the present device has advantages over these methods in its very convenient small size and in the fact that no modifications or internal connections to a generator are required. No feedback to the generator is employed; instead, the generator output is controlled directly.

This development may be considered a "spin-off" from the research activities of the Radio Standards Engineering Division. It was developed for a specific application, in which it performed efficiently. Its users feel that such a device will also be useful in other laboratories dealing with rf signals, in particular standards laboratories of industry and government.

### **Stabilizer Design**

The rf signal stabilizer consists basically of (1) a d-c reference voltage source, (2) a voltage comparison network, (3) a d-c feedback amplifier, and (4) a pair of PIN\* diodes. The reference voltage supply is adjustable

and its setting is dictated by the amplitude of the signal being stabilized. The voltage comparison network detects the peak amplitude of the rf signal, compares it to the d-c reference voltage, and produces a d-c error voltage which is proportional to their difference. This error signal is fed to the feedback amplifier where it is amplified and used as a bias voltage for the PIN diodes. These diodes, which are directly in the rf signal path, act as a voltage dependent attenuator. A d-c regulator circuit is also included for the purpose of supplying the +15 V dc and the -15 V dc required by the d-c feedback amplifier and the d-c reference voltage source.

### **Operational Specifications**

The device fabricated by Mr. Driver is completely solid state and is packaged in a 2x3x6-inch housing. The cost of the electronic parts is approximately \$80.

The stabilizer is capable of handling rf signals having peak voltage levels between 0.5 and 10 volts. Although the device has been optimized for operation at 1 GHz, it can be used, with some limitation, at frequencies as low as 50 MHz. Its stabilizing factor at 1 GHz is 1000 to 1; that is, voltage variations in the incoming rf signal are reduced by a factor of 1000 at the output of the stabilizer. At 1 GHz the insertion loss is less than 2 dB and the dynamic range is approximately 10 dB. The stabilizer requires a d-c input of 70 mA at 50 V. Circuit diagrams and mechanical drawings are available upon request from L. D. Driver at the NBS Radio Standards Engineering Division, Boulder, Colo.

\*Consisting of a p-type, an intrinsic (equally populated by n- and p-type carriers), and an n-type layer.

# USASI COMMITTEE A62 APPROVES A SYSTEMS MODULE STANDARD FOR BUILDINGS

Realization of greater efficiencies and increased economies in the U.S. building industry came one step nearer reality with the recent Committee approval of a standard whose application would eliminate much of the on-site cutting and fitting of building materials. A subcommittee of the United States of America Standards Institute Standards Committee A62 drafted this standard, which establishes a range of coordinated dimensions to control horizontal sizes of building components and systems. At present, USASI Standards Committee A62 has approved the standard by a consensus of its members and has submitted it to the USASI Construction Standards Board for review of its acceptability as a USA Standard, entitled "Standards for a Major Systems Planning and Coordination Module for Building Systems and Components."

This significant contribution to building technology is the product of an industry-government cooperative effort, which is slightly more than 2 years old.<sup>1</sup> USASI Standards Committee A62, entitled Pre-coordination of Building Components and Systems, operates under the national consensus procedures of the USA Standards Institute. The committee activities involve the voluntary participation of 30 major trade associations, over 20 major corporations, and several Federal agencies. It is under the administrative and technical sponsorship of the National Bureau of Standards. Jack E. Gaston of the Armstrong Cork Co. is the chairman.

In the drafting of this standard, the National Bureau of Standards provided support under the direction of Russell W. Smith, Jr. (NBS Building Research Division); and many industry personnel contributed their time

and technical ability. The subcommittee that drafted the standard had the task of devising a dimensional discipline that would foster dimensionally compatible and interchangeable building systems and components without restraining design flexibility or freedom of choice. The subcommittee was chaired by W. Burr Bennett of the Portland Cement Association; other members were:

R. E. Cumrine, National Council of Schoolhouse Construction  
H. F. Hann, Sears, Roebuck & Co.  
George Hanson, Sallada & Hanson  
D. E. Morgenroth, Owens-Corning Fiberglas  
G. J. Murray, American Iron and Steel Institute  
Leonard Pearlmuter, Prestressed Concrete of Colorado, Inc.  
W. K. Platt, American Telephone & Telegraph Co.  
J. W. Glaser, The E. F. Hauserman Co.  
M. K. Snyder, Butler Manufacturing Co.

In the next decade, housing expenditures are expected to range from \$100 to \$200 billion. Studies of building practices in the cutting and fitting of materials show an average loss of construction time of about 5 percent; for some materials the time loss runs as high as 45 percent. By providing a system of coordinated dimensions to eliminate much of the on-site cutting and fitting, this standard should provide obvious economic benefits. In addition, five or six other standards under development could have equal or greater impact.

These additional standards deal with fundamental industrial requirements such as coordination principles and applications, tolerances and fits, joining techniques and detail, and

performance compatibility. The standards developed by Committee A62 will provide the industrial basis for attaining both functional and dimensional compatibility and interchangeability of building components so they can be integrated with a minimum of on-site modification. These guidelines will provide the coordinating control of dimensions, tolerances, and quality so necessary to industrial efficiencies.

A primary appeal of the coordination standards being developed is that they seek to establish basic rules for the application of industrial efficiencies to all construction: both conventional and innovative, on site or in a factory. The standards are not intended to be rigidly applied to every facet of construction, but to be available as a basis for coordination where and when industrialization is desired.

Because such standards are time consuming and difficult to develop and as the benefits are so broad-based, there is little incentive for a single company or industry to invest the necessary effort and money. This is why industry's voluntary standards programs have not generated such standards in the past.

While insufficient individual incentive exists for industry to initiate such standards development on its own, industry has always recognized the advantages of such standards. Thus, when the Commerce Department, through NBS, offered to organize and administer the effort and to provide technical support, an immediate and enthusiastic response was received from the entire industry.

<sup>1</sup> Committee to aid in pre-coordination of building components and systems, NBS Tech. News Bull. 50, No. 10, 176 (October 1966).

*W. P. Harris makes dielectric measurements on a specimen with the ultralow-frequency bridge.*



## VERSATILE ULTRALOW- FREQUENCY BRIDGE CONSTRUCTED

*For  
Dielectric  
and Impedance  
Measurements*

A versatile ultralow-frequency bridge has recently been designed and built by W. P. Harris of the NBS Institute for Materials Research. His work, sponsored by the National Aeronautics and Space Administration, has resulted in a device capable of making complex impedance measurements (magnitude and phase) in the frequency range from 0.001 to 10 000 Hz accurate to within 0.1 percent in magnitude and  $10^{-5}$  radian in phase. Impedances ranging from pure capacitive to pure resistive are easily and quickly measurable.

The bridge is being used at NBS to make high-precision dielectric measurements on new polymer materials. These measurements are important for electrical insulation application, standard reference materials development, and research aimed at a better understanding of molecular behavior and interactions in materials.

The bridge consists basically of three main components: a source, the bridge network, and a detector. Commercial equipment is available for construction of the bridge. Custom parts, however, can be added for increased convenience.

The bridge requires a source that provides both sine and cosine waves, as well as the negative of each. In op-

eration, the positive sine wave (+V) is connected to a cell containing the specimen with capacitance  $C_X$  and conductance  $G_X$ . The negative sine wave (-V) is connected to a variable capacitor ( $C_B$ ) which is adjusted to balance the capacitance of the unknown. The conductance is balanced by feeding the cosine wave\* through another variable capacitor ( $C_G$ ).

Thus, balance equations are extremely simple ( $C_X = C_B$ ). That is, the unknown capacitance is determined from the capacitance required to produce a null on a detector. The dielectric constant is then obtained from  $\frac{C_X}{C_0}$  where  $C_0$  is the empty cell capacitance.\*\*

The dissipation factor, also called  $\tan \delta$ , is given by  $a \frac{C_G}{C_X}$  where  $a$  is the

voltage ratio of  $\frac{|V| \text{ cosine phase}}{|V| \text{ sine phase}}$

The conductance of the unknown,  $G_X$ , is given by

$$G_X = a \omega C_G,$$

where  $\omega$  is the angular frequency. It can be seen that varying  $a$  is equivalent to varying  $C_G$ , and a change of  $a$  by a factor of 10 can be substituted for a change of range of  $C_G$ . Changes



f a are quite practical through the use of precision amplifiers or attenuators in the signal channel.

The bridge network is simply a combination of several capacitors with their low terminals tied together. This common point then becomes the input to the detector. Also connected to this detector terminal is the low side of the cell or specimen holder.

Devices termed as "electrometers" are suitable as detectors in this apparatus. However, some devices that do not fit the descriptions of "electrometers" will also serve. The essential component needed for the detector is a high-impedance voltmeter or charge meter. Certain commercially available operational amplifiers also serve well for the detector. In these devices, input impedances of  $10^{12}$  to  $10^{14}$  ohms are achieved by employing the so-called "electrometer connection," a connection for producing a +1 amplification.

At ultralow frequencies, an X-Y recorder or equivalent is necessary to obtain data. A better solution, however, is the recently introduced variable-persistence oscilloscope, with suitable modifications. This device can serve over the entire frequency range, thus adding a measure of convenience. With the oscilloscope, a Lissajous loop display provides a desirable final detector stage, especially if the phase of the horizontal drive signal is adjustable, so that the loop is phase-sensitive. This allows adjustment for capacitive unbalance in one manner (for example, by tilting the axis of the Lissajous loop) while an unbalance in the conductance produces the opposite response (a change in area of the loop). This requires a third, adjustable phase, output from the source. It can be obtained by employing a commercially available function generator, triggered by the primary signal.

\*For very low loss specimens, the negative of the cosine wave is sometimes required, because of phase shifts in the circuitry.

\*\*This relationship is strictly true only for the case where the dielectric fills all the space between the electrodes, as with a fluid. For solids, some cell factors or corrections must be applied, based on the geometry of the specimen and cell.

## KUSHNER HEADS INSTITUTE FOR APPLIED TECHNOLOGY

Lawrence M. Kushner has been named Director of the NBS Institute for Applied Technology. Dr. Kushner had been Acting Chief of the Institute since last May. He replaces John P. Eberhard, who resigned to join the University of New York at Buffalo. Dr. Kushner had been Deputy to Mr. Eberhard since 1966.

The Institute for Applied Technology has a range of programs aimed at facilitating the use of new technology in Government and industry. One of the Institute's main tasks is to develop standards and methods for measuring the properties of the sophisticated materials and manufactured products of modern technology. This technological standards work is the basis for innovation and quality control in a wide variety of industries. Among the major units in the Institute are those concerned with materials evaluation, weights and measures, invention and innovation, vehicle systems, building research, electronic instrumentation, and technical analysis. Dr. Kushner will also have under his general direction the Clearinghouse for Federal Scientific and Technical Information and the Center for Computer Sciences and Technology.

Before becoming Deputy Director of the Institute for Applied Technology, Dr. Kushner was Chief of the NBS Metallurgy Division from 1961 to 1966. In 1964 and 1965 Dr. Kushner was a Commerce Science and Technology Fellow working in the office of Assistant Secretary of Commerce J. Herbert Hollomon as a Special Assistant for Legislation. Dr. Kushner joined the Bureau in 1948 as a physical chemist in the Surface Chemistry Section, later becoming Assistant Chief of the Section. In 1956 he was appointed Chief of the Metal Physics Section. Dr. Kushner's main scientific research interests were in the relation of defects in the crystal structure of metals to the metal's physical properties.

Dr. Kushner was graduated from Queens College with a B.S. in chemistry in 1945. He received both his A.M. (1947) and Ph.D. (1949) in physical chemistry from Princeton University.

# CONFERENCE & PUBLICATION *Briefs*

## DENTAL RESEARCH SYMPOSIUM

A Symposium celebrating the 50th anniversary of the NBS Dental Research Section will be held at the Gaithersburg (Md.) facilities of the National Bureau of Standards from October 6 to 8, 1969.

The program will recognize the achievements in the dental research field over the last half century, with emphasis on the research program carried on at NBS in cooperation with the American Dental Association, Federal Government agencies, and industrial laboratories. The conference will also present a comprehensive state-of-the-art report and a look at present trends and future expectations in the field of dental materials. Papers will be presented by outstanding authorities in the dental field, both national and international, in addition to those by the NBS staff.

The Symposium will be sponsored jointly by NBS and the American Dental Association. For further information, write to:

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Rm. A145, Polymers Bldg.  
National Bureau of Standards  
Washington, D.C. 20234

## APPLYING NEW MEASUREMENT TECHNIQUES TO STUDY DRUG METABOLISM

NBS acted as host to more than 400 biologists, chemists, pharmacologists, and other scientists who attended the Conference on Applications of Newer Physical Techniques to the Study of Drug Metabolism June 12-14, 1968, at the Bureau's new laboratory complex in Gaithersburg, Md. The conference was jointly sponsored by the National Academy of Sciences-National Research Council, the National Institutes of Health, the National Bureau of Standards, and the Pharmaceutical Manufacturers Association Foundation, Inc.

The ingenuity of the organic chemist has led to the introduction of many new drugs and other chemicals into our environment. Difficult problems have been encountered in the study of these foreign substances because they are present at exceptionally low concentrations in biological materials.

Fortunately, considerable technical advance has been made in analytical methodology during the past decade. Instead of detecting and characterizing foreign substances in biomaterials at the microgram level, it is now possible to measure nanograms of these trace constituents.

The purpose of the conference was to familiarize researchers in the biological sciences with the potentialities

and limitations of the newer physical techniques that have been developed for the study of the metabolism of foreign substances. How these techniques have been used to separate, identify, and measure the quantity of drugs and other foreign substances in biological materials was emphasized by the conference speakers. Unlike other conferences dealing with new instrumental techniques, this conference stressed their biological applications.

The conference was opened with welcoming remarks by Dr. C. L. Dunham, Chairman of the Division of Medical Sciences, NAS-NRC; Dr. F. L. Stone, Director of the National Institute of General Medical Sciences, NIH; and Dr. A. V. Astin, NBS Director. Most of the papers described a particular technique and were followed by either a question period or a discussion of the applications of the technique.

Several sessions were devoted to gas-liquid chromatography (GLC). These covered general principles of GLC, GLC detectors, derivative formation for GLC (in general and by electron capture detection), and pyrolysis in GLC. Three other sessions were panel discussions on the facilities and personnel required for a physical chemistry laboratory to study drug metabolism, the training of such personnel, and the needs for further research on new physical techniques.

Other sessions covered separation techniques, fluorescence spectrometry, phosphorescence spectrometry, radioactive derivative techniques, oscillographic polarography, x-ray diffractometry, nuclear magnetic and electron paramagnetic resonance, structure determination of drug receptor complexes by NMR, and integrated systems of mass spectrometry.

The role that NBS can play and is playing in the area of biological sciences is that of providing advanced capability and techniques for meaningful measurement as well as standard reference materials. W. W. Meinke, Chief of both the NBS Analytical Chemistry Division and the NBS Office of Standard Reference Materials, covered the Bureau's activities in these areas by reviewing examples of analytical competences such as electroanalytical, gas analytical, mass analytical, microchemical, nuclear resonance, and optical methods, which are being brought to bear on biomedical problems.

Dr. Meinke reviewed the Bureau's work in dental materials and standard reference materials that have biological applications. Of the more than 600 standard reference materials available from NBS, over 50 certified standards are of direct use to health programs, and more than 50 others in this category are currently being prepared or planned. Those standards already available include a

cholesterol standard, a number of radioisotopes and labelled sugars, a nicotinic acid microchemical standard, and 3 gaseous standards of carbon dioxide in air.

The conference proceedings will not be published; however, the Conference is one of an extensive series from which the Committee on Problems of Drug Safety of the NAS-NRC will derive source material for the ultimate publication of a series of papers on methods of drug evaluation.

Mr. Duke C. Trexler, Executive Secretary of the NAS-NRC Drug Research Board, is primarily responsible for organizing this series of conferences. The Chairman of the Committee on Problems of Drug Safety is Dr. J. J. Burns, NAS-NRC; and Dr. G. J. Mannering, Department of Pharmacology, University of Minnesota, was the Director of this Conference.

### FIFTY-THIRD NATIONAL CONFERENCE ON WEIGHTS AND MEASURES

The Fifty-Third National Conference on Weights and Measures was held June 17-21 at the Sheraton-Park Hotel in Washington, D.C. More than 500 representatives—State and local weights and measures officials, Federal officials, and representatives from consumers, industry, and business—were in attendance.

This year's Conference had a strong consumer orientation; one of its most important actions was the adoption of a new Model State Packaging Regulation. The promulgation of packaging and labeling regulations by the Food and Drug Administration and the Federal Trade Commission, in line with their responsibilities under the Fair Packaging and Labeling Act, necessitated a complete rewriting of the Model State Regulation pertaining to packages.

Founded and sponsored by the National Bureau of Standards, the Conference is one of the principal forums for discussion of consumer affairs in the Nation. It is a meeting ground for all levels of government concerned with weights and measures administration, industries associated with weighing and measuring equipment and packaging, and industries that offer their products for sale in weighed and measured quantities. The concern of the Conference is in protecting both buyer and seller. To this end, the Conference considers weights and measures laws, regulators, and technology.

L. C. McQuade, Assistant Secretary of Commerce for Domestic and International Business, was one of the principal speakers on the Conference program. He explained the role of the Department of Commerce in relation to the national economy.

The Assistant Secretary was followed by A. V. Astin, NBS Director and President of the Conference. Dr. Astin spoke on the responsibilities of the Bureau in its leadership role in the National Measurement System and in facilitating applications of technology to the achievement of national goals.

Conference Chairman C. C. Morgan addressed the general session; he explained that while the National Conference on Weights and Measures has no regulatory authority, State regulation of weights and measures could bring chaos in interstate commerce without the Conference's unifying presence. "The Conference," he emphasized, "is dedicated to the promotion of the principles of fairness, impartiality, uniformity, and equity in all matters pertaining to weights and measures administration."

Many interesting and informative talks were given during the Conference on such topics as label manufacturing under the Fair Packaging and Labeling Act, weights and measures in Ceylon, and packaging standards for fluid milk products.

The delegates and guests attended a Conference luncheon on Wednesday, June 17. In an address following the luncheon Congressman J. T. Myers of Indiana described the increase in the usage of the metric system throughout the Nation and the world and spoke of the need for a study to determine the problems of converting to the metric system in the United States.

Other topics covered in the Conference included how to get the most efficiency in weights and measures administration when confronted with limited resources, metering technology in the petroleum industry, and the development of a State Measurement Center.

### INSULATING GLASS SEMINAR

NBS, in association with the American Society for Testing and Materials, will sponsor a seminar on the Durability of Insulating Glass Units November 14 and 15, 1968, at its facilities in Gaithersburg, Md.

Insulating glass units in windows can mean many things to building occupants—thermal comfort, better humidity control, reduced noise, enhanced privacy, and a clear view of the world. A reliable insulating glass assists materially in the economical heating and cooling of homes, offices, schools, and work places, and in reduced maintenance costs. At present more than 75 American and 25 Canadian firms manufacture insulating glass units—evidence that this product has come of age.

The conference is specifically designed to provide information that will lead to better insulating glass performance. It is open to any one concerned with the design, manufacture, specification, purchase, installation, or maintenance of windows. Technical papers and panel discussions by experienced professional, governmental, manufacturing, and installation authorities will provide practical solutions to the following questions, among others: What do 5-, 10-, or 20-year guarantees mean? Are guarantees indicative of actual performance in service? How do manufacturers control production? Can independent laboratories check the product's performance before trouble arises? In addition, new test methods and performance standards will be proposed and discussed. Foundations will also be laid for a performance certification

*continued on page 215*

# REFERENCE BASE OF THE VOLT TO BE CHANGED



*Computable capacitance standard  
recent absolute definition of  
The capacitance standard is  
terms of its length, the cap-  
of the standard cannot be bal-  
can be balanced against a resis-  
value of the resistors.*

On January 1, 1969, the National Bureau of Standards expects to introduce a new base of reference for the volt, the practical unit of electromotive force (emf), or voltage. Under this change, the value of the U.S. legal volt, as maintained by a group of standard cells at NBS, will differ by about 10 parts per million from its previous value. The new value represents a better measurement of the voltage of these cells in terms of the theoretical unit of emf derived from the basic mechanical units of length (meter), mass (kilogram), and time (second).

The proposed action by NBS will be coordinated with planned international action to bring the volt units of 10 countries into agreement. This action is expected to be taken by the International Committee on Weights and Measures, at its meeting during October 1968, on the recommendation of its Advisory Committee for Electricity. It will be the first change in the volt that the International Committee

has made since 1946, when it recommended a conversion from the "international" electrical units to the "absolute" system.\* The recommendation of 1946 was universally adopted on January 1, 1948 [1, 2, 3].\*\*

The expected change in the U.S. volt is being announced at this time so that those who provide calibration services for standard cells and who issue calibration reports can make preliminary plans for the necessary changes in their reports, pending the official action on the change in the base of reference for the volt. This announcement is also being made so that those who are engaged in work of the highest precision, such as determinations of physical constants which directly or indirectly involve electrical units, may have advance knowledge of the change. In calibration reports issued by NBS during 1969, values will be given on both the old and the new basis.

At its meeting in Paris in October 1946, the International Committee

on Weights and Measures accepted the relation:

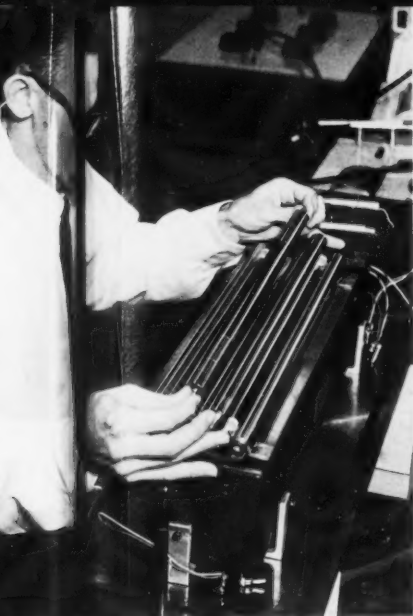
$$1 \text{ mean international volt} = 1.000\,34 \text{ absolute volts.}$$

The mean international volt to which this equation referred was the average of the units maintained in the national laboratories of the six countries—France, Germany, Great Britain, Japan, the Soviet Union, and the United States—which took part in this work before World War II. The unit of emf maintained by NBS at that time differed from this average by 10 ppm, as evidenced by the international comparisons made at the Bureau International des Poids et Mesures (BIPM), Sèvres, France. Accordingly, the conversion factor for the volt adopted in the U.S. on January 1, 1948, was

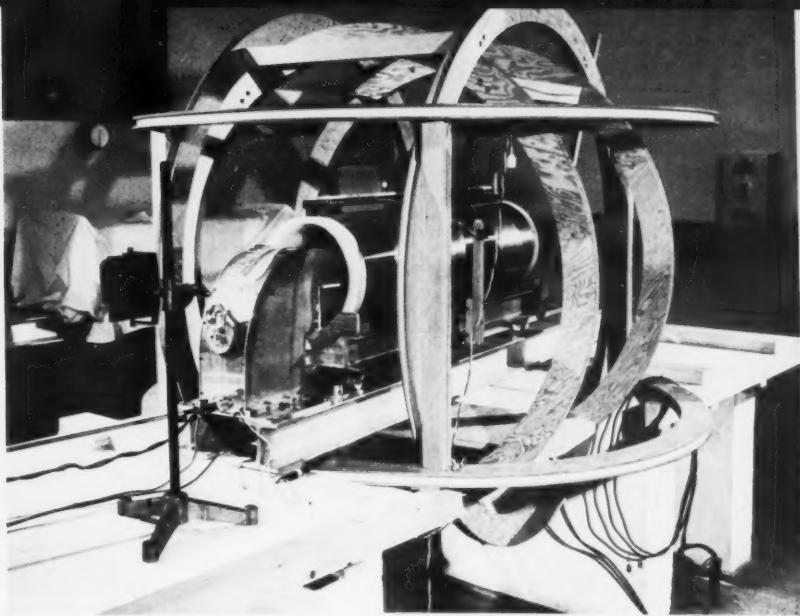
$$1 \text{ international volt (U.S.)} = 1.000\,33 \text{ absolute volts.}$$

The new change will be considerably smaller than the one of 1948. It will mean that





able capacitor standard used in the most absolute determination of the NBS ohm. The capacitance standard is computable in terms of its length, the capacitive reactance standard against a known frequency, and a balanced resistance to give the value of the resistance.



Electrodynamometer used in the most recent absolute determination of the NBS ampere. The metal housing that enters the large solenoid contains a small rotatable coil and a balance arm. When the direction of the current through the coils is reversed, a torque is produced on the small coil. This coil is connected to the balance arm so that as it tends to rotate, equilibrium is upset. Balance is restored by adding known weights to the balance arm by means of the rod-and-pulley arrangement at the outer end of the housing. From the known weight, the length of the balance arm, and the geometry of the windings, the value of the current in amperes can be calculated.

1 present volt (U.S.) will be about 1.000 010 new volt (U.S.).

This factor does not represent an adjustment for a drift in the U.S. emf standard, i.e., for a drift in the emf of the group of standard cells used to maintain the unit. Rather it represents the improvement since World War II in the accuracy with which the voltage of these cells can be measured in terms of the absolute volt.

The factor required to bring the volt unit into international agreement will differ only slightly among the ten countries—Australia, Canada, France, Germany (East), Germany (West), Great Britain, Italy, Japan, the Soviet Union, and the United States—which now participate every third year in international comparisons of standard cells and standard resistors at BIPM. The International Bureau has ascertained these small differences by intercomparing the values of the standard cells and standard resistors that define the units of

current of the various nations in terms of the BIPM ampere [4].

The increasing importance of precision measurements has made it evident that a more accurate realization of the defined volt is needed. New measurements of the ohm and the ampere in terms of the basic mechanical units (absolute measurements) have been made in various national laboratories [5, 6, 7]. The ohm determinations show that the values of the standard resistors used to maintain the unit of resistance are substantially correct (to better than 1 ppm) in terms of the basic mechanical units. On the other hand, the recent ampere determinations indicate that the absolute ampere is smaller by about 11 ppm than the ampere determined by the standard cells and standard resistors used to maintain the standards for the volt and ohm, respectively, at BIPM. Since these measurements indicate that no adjustment in the ohm is called for, it is evident that an adjustment in the base of reference

for the volt is needed to account for the results obtained on the ampere.

The recent absolute measurements at NBS include an ohm determination in terms of a computable capacitor, and three ampere determinations, one in 1958 with the NBS current balance [8] and two with a Pellat-type dynamometer [9]. The results of the latest ampere determinations, not yet published, are in excellent agreement with the 1958 current balance value [8]. Since an ampere determination necessarily involves a force measurement, the final assignment of the ampere value must take account of recent determinations of the acceleration due to gravity, one of which was made at NBS [10, 11, 12].

Final announcement of the actual factor to be used in defining the new base of reference for the volt will be made after the International Committee meeting in October. Selected references for more details on the base of reference for the volt and related electrical quantities are given below.

*continued*

## VOLT *continued*

1. Curtis, H. L., **Review of recent absolute determinations of the ohm and ampere**, J. Res. NBS 33, 235 (1944); gives history leading to 1948 conversions from "international" electrical units to "absolute" electrical units.

2. **Announcement of Changes in Electrical and Photometric Units**, NBS Circ. C459 (1947).

3. **Changes in electrical and**

**photometric units**, NBS Tech. News Bull. 31, No. 5, 49 (1947).

4. Terrien, J., **The work of the Bureau International des Poids et Mesures concerning electromagnetic units and measurements**, IEEE Trans. Instr. Meas. IM-15, No. 4, 140 (1966).

5. Vigoureux, P., **A determination of the ampere**, Metrologia 1, No. 1 (1965).

6. Rayner, G. H., **An absolute determination of resistance by**

**Campbell's method**, Metrologia 3, No. 1 (1967).

7. Thompson, A. M., **An absolute determination of resistance based on a calculable standard of capacitance**, Metrologia 4, No. 1 (1968).

8. Driscoll, R. L., and Cutkosky, R. D., **Measurement of current with the National Bureau of Standards current balance**, J. Res. NBS 60, 297 (1958).

9. Driscoll, R. L., **Measurement of current with a Pellat-type electro-dynamometer**, J. Res. NBS 60, 287 (1958).

10. Tate, D. R., **Absolute value of  $g$  at the National Bureau of Standards**, J. Res. NBS 70C (Engr. and Instr.), No. 2, 149 (1966).

11. **Acceleration due to gravity measured at NBS**, NBS Tech. News Bull. 51, No. 3, 50 (Mar. 1947).

12. Tate, D. R., **Acceleration due to gravity at the National Bureau of Standards**, J. Res. NBS 72C (Engr. and Instr.), No. 1, 1 (1968).

13. Cutkosky, R. D., **Evaluation of the NBS unit of resistance based on a computable capacitor**, J. Res. NBS 65A (Phys. and Chem.), 147 (1961).

14. Hamer, W. J., **Standard Cells, Their Construction, Maintenance, and Characteristics**, NBS Mono. 84 (1965); includes history of volt units and international comparisons of standard cells.

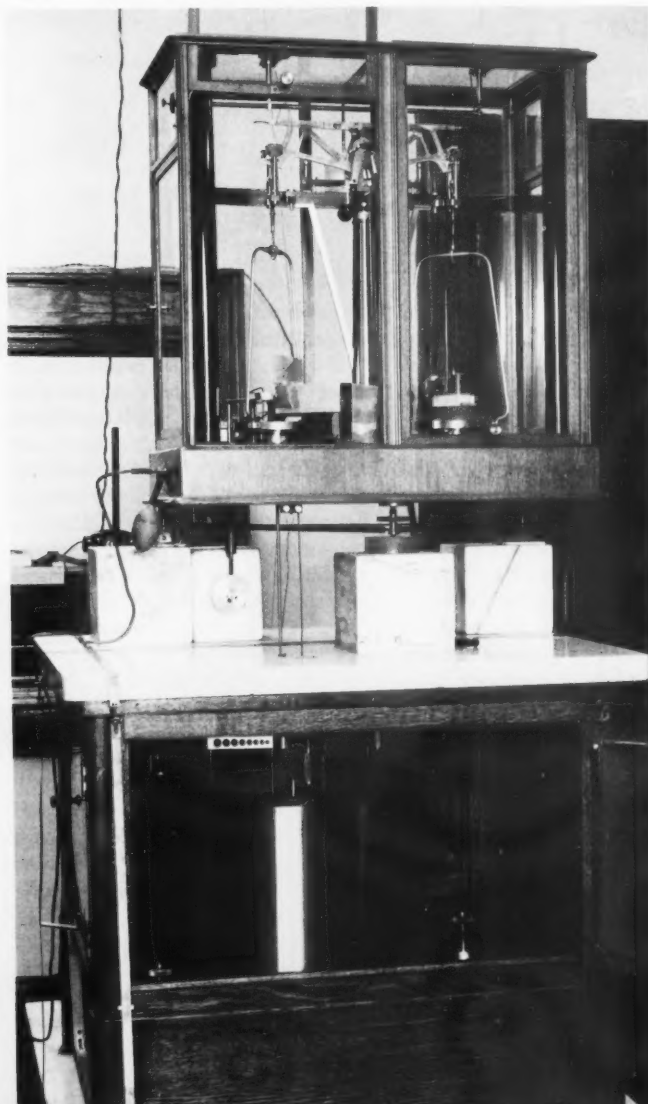
15. Hamer, W. J., **History of the National Standard of Electromotive Force**, ISA, M2-1-Mestind-67 (1967).

16. Hamer, W. J., **A Joule centennial**, J. Chem. Ed. 45, 123 (1968); gives early history of relation between B.A. and cgs units of resistance.

\*The "international" units in use before 1948 were defined in terms of the so-called "reproducible" units adopted internationally near the turn of the century—the ohm, defined as the resistance of a specified mercury column; and the ampere, defined as the current that would deposit silver at a certain rate under specified conditions. These units should not be confused with the present "absolute" units of the "Système International" (international system), which are defined in terms of the meter, kilogram, second, and an assigned value of space permeability (magnetic constant),  $4\pi \times 10^{-7}$  henry per meter.

\*\*Figures in brackets refer to references at the end of the article.

*Current balance on which the Bureau made an absolute measurement of the ampere in 1958. In this apparatus the force between two wires carrying current is balanced against the gravitational force of a known mass.*



# REFRACTIVE INDEX OF HYDROGEN MEASURED

Highly precise values of the refractive indices of gaseous and liquid hydrogen have been obtained by D. E. Diller<sup>1</sup> of the NBS Institute for Basic Standards. This research was carried out to obtain information for use in determining fluid densities and mass-flow rates of parahydrogen—a propellant now being used extensively in advanced U.S. space programs. The work was part of a major program of research, sponsored by the National Aeronautics and Space Administration, through its Space Nuclear Propulsion Office, on the thermodynamic and transport properties of parahydrogen.

One of the main objectives of the NBS refractive index study was to provide highly accurate, self-consistent data that would make similar researches unnecessary in other laboratories. Another objective was to examine and analyze the density and temperature dependence of the Lorentz-Lorenz function. A basic theory, advanced by H. A. Lorentz, predicts that the function,  $\frac{n^2 - 1}{n^2 + 2} \cdot \frac{1}{\rho}$ , where  $n$  is the index of refraction and  $\rho$  is the density, is a constant; that is, its value should be nearly independent of density and temperature for a nonpolar substance such as hydrogen.

To obtain refractive index measurements with the necessary precision to fulfill these objectives, a small Fabry-Perot interferometer was constructed. This interferometer consists essentially of two plane parallel semitransparent surfaces, separated by a spacer of accurately known length. One advantage of this apparatus is that the interferometer can be placed entirely within the sample holder, thus minimizing density gradients and eliminating distortions due to pressure changes.

Illumination for the interferometer was an extended source of nearly monochromatic 5462 Å radiation, ob-

tained by passing light from a mercury-198 lamp through a green narrow-band interference filter. When this light is passed through the interferometer, a circular fringe pattern is formed.

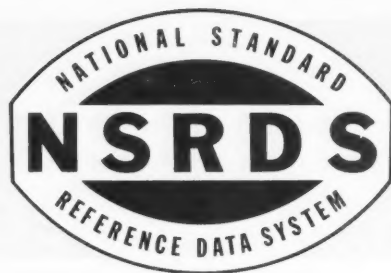
After the hydrogen sample was admitted to the interferometer, continuous measurements were made while the sample was attenuated from high pressure to vacuum. During this process, the intensity of the center of the fringe pattern, which changed with the density and refractive index of the sample, was recorded, and the number of fringes that passed a reference point was counted.

These data were then translated into absolute refractive indices by using a formula that took into account the laboratory measurement of the fringe count, the spacer length in vacuum, and the fluid pressure. It is of interest to note that this was the first time that absolute refractive index measurements of a liquid have been made using an interferometric method.

During the NBS study, some 200 refractive index measurements were made on normal and parahydrogen at temperatures between 15.0 and 298.15 K, and at pressures up to 233 bars. The densities ranged from 0.002 to 0.080 g/cm<sup>3</sup>, and the refractive indices ranged from 1.003 to 1.125.

From these measurements, the Lorentz-Lorenz function was computed for a wide range of temperature-density combinations. One striking feature of the results is that the Lorentz-Lorenz function for parahydrogen was found to change by only 0.5 percent over a very large range of these parameters. It was possible, however, to show that real temperature and density dependences do exist for this function.

<sup>1</sup> Diller, D. E., Measurements of the refractive index of gaseous and liquid hydrogen, *J. Chem. Phys.* (to be published).



# NEWS

*This column regularly reports significant developments in the program of the National Standard Reference Data System. The NSRDS was established in 1963 by the President's Office of Science and Technology to make critically evaluated data in the physical sciences available to science and technology on a national basis. The System is administered and coordinated by the National Bureau of Standards through the NBS Office of Standard Reference Data, located in the Administration Building at the NBS Gaithersburg Laboratories.*

## NSRDS Status Report

NBS Technical Note 448, *Status Report—National Standard Reference Data System, April 1968*<sup>1</sup> (70 cents), summarizes the status of activities of the Office of Standard Reference Data. The report provides a detailed review of data compilation activities within the program's seven broad categories: nuclear data, atomic and molecular data, solid state data, thermodynamic and transport data, chemical kinetics, colloid and surface properties, and mechanical properties. Progress in data systems design and development and in information services of NSRDS are also reviewed. Certain problem areas of the program are identified in the report. The appendix lists information and data centers associated with the Office of Standard Reference Data, NSRDS publications, and organizations, groups, or individuals compiling or evaluating data.

This publication reports the progress made by the Office of Standard Reference Data in developing NSRDS. Since the program came into existence in 1963, the magnitude of the effort required to implement the System has been estimated more closely, procedures for the establishment of data centers have been developed, and relationships with other agencies have been more clearly defined. The Office has learned that the task of carrying out a critical evaluation is more demanding of both time and intellect than most members of the technical community undertaking these jobs had thought. The tasks involved have been clarified, and the qualifications of the staff needed within the data centers have been better formulated.

The original emphasis of NSRDS was on the production

of compilations of critically evaluated data, with a somewhat lesser emphasis on the production of critical reviews. Operating experience has shown that emphasis must also be placed on critical analysis of the sources of uncertainty in the measurement results reported in the literature. Estimates made of the fraction of the literature in any specific field containing data worthy of a detailed critical evaluation range from approximately 50 percent down to a low of only 8 or 10 percent. The Report indicates that in almost every field in which a reasonably common measurement technique is employed, criteria need to be developed for the conditions under which experimental measurements should be made. Additional criteria should be developed for reporting experimental results in the literature. Procedures are needed to ensure that results of quantitative measurements are made available to the data analysis center, even though they may not be reported in formal archival literature. In each of the fields in which the Office is active, the Report states that these needs will be examined in detail.

NSRDS progress has been slower than desirable; initial plans called for much more rapid development. The rate of development has, of course, been determined by the System's financial resources, which have been only a relatively small fraction of the estimated amount needed to accomplish the desired task expeditiously. Nevertheless, many useful products have appeared. When this Status Report was written, 31 separate compilations and 6 critical reviews had been published or were in press; 16 non-critical compilations or bibliographies have appeared. Others are in various stages of preparation.

As these products appear, the Report states, greater attention is being paid to the general problem of making them as readily accessible to the technical community as possible. In business terms, this is a marketing problem. The Office of Standard Reference Data has asked the question, "How should we market our products and services in order to accomplish the most good for the progress of science and technology in the United States?" The Office believes that a variety of marketing procedures must be employed, and it is gathering the information needed to develop plans for operation.



## PRECISE: A Multiple Precision Version of Omnitab

One of the more troublesome problems that confront the careful user of modern computers is the loss of significance resulting from round off and other computing pitfalls. In many calculations rounding errors are serious sources of annoyance—in some they throw the results completely off. While the recent trend to design computers with built-in hardware for double-precision operations is a decided help in this regard, the user of these features must still be on guard. He must guard against possible flaws in the hardware or algorithms, and even, unhappily, against errors in important constants used by the compiler or the conversion routines.

The problem has become more serious recently because many of the third generation computers have a shorter word length. As a result, programs which previously gave suitable answers in single precision now must be run in double precision.

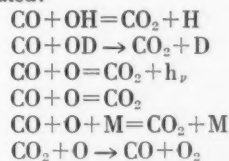
Soon after the proving out of the Omnitab<sup>2</sup> general-purpose computing program, effort at NBS was turned to the design of a comparable system for more precise calculations than were then possible in single precision. This system drew heavily on the multiple precision package previously designed by Alfred E. Beam of the University of Maryland. This package spares professional programmers the tedium of writing painstaking instructions for the computer to handle double and triple precision and out-of-range arithmetic. NBS Technical Note 446, *PRECISE: A Multiple Precision Version of Omnitab*<sup>1</sup> (55 cents), by Alfred E. Beam and Joseph Hilsenrath, describes how the package has been used to provide nonprogrammers with a computer tool for very precise calculations without resorting to conventional, tedious programming.

The PRECISE program was designed to carry out arithmetic operations and function generation, often to as many as 28 significant digits and at the very least to 21 figures. Except when instructed to increase the ranges, the program normally handles any number in the range  $10 \exp(-76)$  to  $10 \exp(76)$  and gives results to 28 significant figures. The program can also handle numbers outside of the above range. The greatest or smallest power of 10 can be as high as plus or minus one billion. In this extreme case the results are good only to 21 significant figures.

### High Temperature Rate Data

The Department of Physical Chemistry, The University, Leeds, England, has just issued the report, "Critical Evaluation of Rate Data for Homogeneous, Gas-Phase Reactions of Interest in High-Temperature Systems," by D. L. Baulch, D. D. Drysdale, and A. C. Lloyd. In this report the rate data are tabulated, paper by paper, together with an indication of the method used and the evaluators' comments. These data and the recommended rate coefficient are also graphed.

The available rate coefficient data for the following reactions are evaluated:



A discussion and a bibliography are provided for each reaction.

This work is part of the High Temperature Rate Data Project supported by the U.K. Office of Scientific and Technical Information, which administers the British Data and Information Program. This Office is the U.K. equivalent to the NBS Office of Standard Reference Data, which administers the U.S. NSRDS program. Further information about the work of the Leeds Group or copies of reports may be obtained from Dr. D. L. Baulch, School of Chemistry, The University, Leeds, 2, United Kingdom.

### Nuclear Magnetism to be Reviewed

L. H. Bennett, Director, Alloy Data Center and Chief, NBS Alloy Physics Section, has been selected to write Chapter 14 on "Nuclear Magnetism" in the 1968 *Magnetism and Magnetic Materials Digest*. The *Digest* is published annually and contains a survey of the technical literature of the preceding year. *Magnetism and Magnetic Materials—1968 Digest* will contain 21 chapters of general subjects such as Neutron Diffraction, Spin Configuration and Magnetic Transitions, Properties of Non-Metals, Thin Films, and Magnetic Domains. Each chapter will consist of a bibliography, materials index, and a brief, noncritical evaluation of all papers published during the previous year within the scope of each author's chapter. Dr. Bennett's chapter will deal with all papers published in 1967 relating to nuclear magnetism in exchange-coupled electron-magnetic materials. Some of the topics included are: Hyperfine interactions, nuclear specific heat, Mössbauer effect, nuclear magnetic resonance, Knight shift, s-d interaction, and temperature dependence of spontaneous magnetization. Approximately 185 papers will be reviewed in this chapter alone.

The 1968 *Digest* will be published by the Academic Press, and will be sold at the 14th Annual Conference of Magnetism and Magnetic Materials in November 1968.

### Photoabsorption Cross Section Data

JILA Information Center Report No. 5, "Bibliography of Photoabsorption Cross Section Data," by L. J. Kieffer, provides references to published low energy atomic collision data dealing with cross sections of absorption and ionization coefficients for wavelength regions of continuous absorption. The bibliography is divided into three main sections. The first section describes the data under references according to a hierarchy of descriptors. The second section lists title, authors, and complete refer-

*continued*

ences for the papers cited. The third section is an alphabetical author index. Copies of the bibliography are available from the JILA Information Center, University of Colorado, Boulder, Colo. 80302.

### AMPIC Issues New Bibliography

ORNL-AMPIC-10, *Bibliography of Atomic and Molecular Processes for July-December 1967*,<sup>3</sup> published in June 1968, is the eighth in a series of bibliographies published by the Atomic and Molecular Processes Information Center (AMPIC) at Oak Ridge National Laboratory. The Center is jointly sponsored by the U.S. Atomic Energy Commission and the NBS Office of Standard Reference Data. This annotated bibliography contains references of interest to atomic and molecular processes research. Bibliographic sources consist of eighty scientific journals and five abstract journals. As in the previous bibliographies of this series, references are classified into fourteen major categories with appropriate subcategories, and are entered alphabetically in each category with the reactants or the atomic and molecular system of interest.

### Chemical Kinetics

Compilation, evaluation, and tabulation of numerical data in the field of chemical kinetics presents a number of special problems. By definition, the system is changing in composition—the reaction mechanism may be uncertain, or may change with temperature or other parameters; catalysts, wall-effects and other local influences may be significant. Previous attempts to present kinetic data in tabular form have found the difficulties substantial. One such attempt was NBS Circular 510, *Tables of Chemical Kinetics—Homogeneous Reactions*, issued with two supplements, and prepared under the direction of N. Thon and Charles Stauffer.

To obtain advice on a program which might avoid some of these problems, the NBS Office of Standard Reference Data sought the counsel of the NAS-NRC Committee on the Kinetics of Chemical Reactions. The recommendations of the Committee recognized the difficulties inherent in any attempt to evaluate and compile the large amount of numerical data published on chemical reaction rates. The Committee suggested a program of critical review monographs on narrow topics, to be written by qualified specialists. Their purpose would be to provide comprehensive coverage, with emphasis on the quantitative aspects and with extensive bibliographies.

A number of critical reviews have been started and some are completed. Experience to date indicates that the critical review approach is valuable. Additionally, rate constant data were carefully selected for compilation and critical evaluation in areas of bimolecular chemical reactions. These kinetic data have now been published in tabular form. The assembly and publication of data sheets

describing the kinetics of individual chemical reactions are also being encouraged. In all these efforts it has become apparent that the author must be careful to limit the scope of his topic to avoid being overwhelmed by the quantity of literature he must read and analyze.

It has also become clear that both the review authors and the user public can be helped greatly by the establishment of information and data centers which function to:

- (a) Help stimulate qualified prospective authors to critically evaluate kinetic data in which they have expertise;
- (b) provide authors of monographs with bibliographic services and assistance;
- (c) maintain coverage of the scientific literature on a current basis;
- (d) store and classify the data and bibliographic content of critical review monographs;
- (e) provide information to the scientific public on the basis of items (c) and (d).

Two data centers have been established by the Office of Standard Reference Data: the NBS Chemical Kinetics Information Center, acting in a general capacity (*NSRDS News*, September 1966),<sup>4</sup> and the University of Notre Dame Radiation Chemistry Data Center (*NSRDS News*, January 1968).<sup>5</sup> At present, these centers are performing services listed as (b) and (c) above, and are providing some limited information services to the public as well. Establishment of additional centers and arrangements for additional critical review monographs have been hampered by lack of funds. The support of the Department of Defense's Advanced Research Projects Agency has provided means for partial support of the Chemical Kinetics Information Center. The Atomic Energy Commission has shared in supporting the Radiation Chemistry Data Center.

### Reaction Rate Newsletter

Workers in chemical kinetics may be interested in receiving the *Defense Atomic Support Agency Information and Analysis Center Newsletter*, which provides summaries of current progress reports in the reaction rate field. The newsletter contains privileged information which should be considered preliminary in nature and subject to possible change; it should not be cited in any publication without prior approval. Persons wishing to subscribe should send their requests to DASA Information and Analysis Center, General Electric—TEMPO, 816 State St., Santa Barbara, Calif. 93102.

<sup>1</sup> Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for the price indicated.

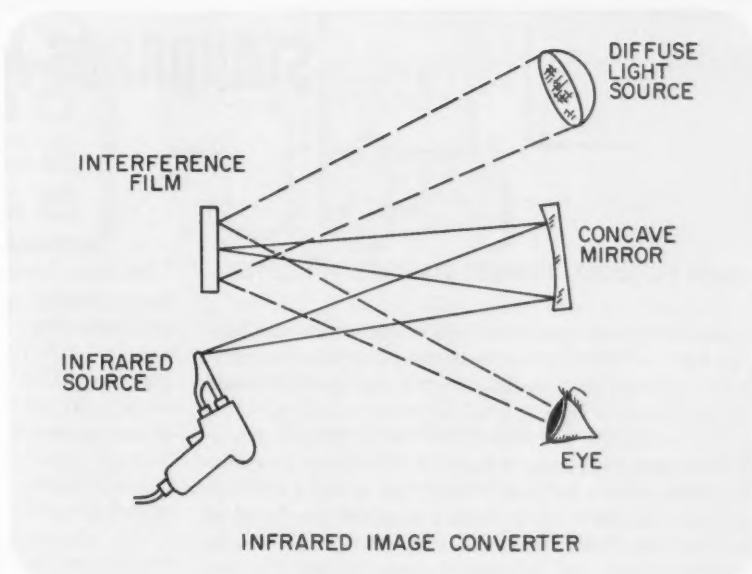
<sup>2</sup> Hilsenrath, J., Ziegler, G. G., Messina, C. G., Walsh, P. J., and Herbols, R. J., *OMNITAB—A Computer Program for Statistical and Numerical Analysis*, NBS Handbook 101, U.S. Government Printing Office, Washington, D.C. 20402, Mar. 4, 1966 (\$3).

<sup>3</sup> Available from the Atomic and Molecular Processes Information Center, Oak Ridge National Laboratory, P.O. Box Y, Oak Ridge, Tenn. 37831.

<sup>4</sup> *NSRDS News*, NBS Tech. News Bull. 50, No. 9, 166-168 (September 1966).

<sup>5</sup> *NSRDS News*, NBS Tech. News Bull. 52, No. 1, 14-16 (January 1968).

*The infrared image converter, or interference film, is the heart of the process that turns infrared into visible images. The film is specially prepared by exposing an emulsion in contact with mercury to laser light.*



## SIMPLE AND INEXPENSIVE INFRARED IMAGE CONVERTER

A new image converter<sup>1</sup> that permits viewing and photographing of normally invisible infrared and ultraviolet radiation was recently devised by C. S. McCamy and J. H. Suter of the NBS Institute for Basic Standards. Objects emitting heat, even though they are not ordinarily visible by their own radiation, can be made visible by the image converter and can be photographed in color. Developed under sponsorship of the Department of Defense, the converter has been successfully used in the laboratory and offers promise of an imaging process that is both simple and economical.

Image converters that change infrared into visible images have widespread applications. They are used, for example, in military night-vision devices and in the medical diagnosis of certain types of cancer. These converters, however, are generally expensive and quite complicated.

In the NBS device, a concave mirror focuses an infrared image on a special plate. This energy changes the color of the plate by changing humidity conditions, thus making the image visible.

The special plate is, in fact, the converter; it is produced by first putting a very fine-grained photographic emulsion in contact with mercury. The emulsion is then exposed, through the glass back on which it is deposited, to light from a helium-neon laser. The light reflected from the mercury combines with light that comes directly from the laser to produce standing waves of light in the emulsion.

When the photographic plate is developed, these standing waves become closely spaced layers of silver grains. The layers give the plate a color that depends on the humidity, changing from red to blue and then to white as the humidity is increased. Absorption of moisture from the air expands the gelatin; this increases the distance between the layers of silver, and as a result produces the color.

When an infrared image is focused on the plate, a diffuse light shining on the plate reveals the infrared image. At this point the image may be seen and can also be photographed.

<sup>1</sup> McCamy, C. S., and Suter, J. H., A new image converter, presented at the Spring Meeting of the Optical Society of America, Washington, D.C., Mar. 13-16, 1968.



# STANDARDS AND CALIBRATION

## HIGH-FREQUENCY CURRENT STANDARD DEVELOPED

Accurate measurements of high-frequency current have long been a difficult problem to the electronics industry. In fact, because such measurements have been so difficult, they have largely been ignored. However, recent advances in high-frequency instrumentation by equipment manufacturers and measurement needs of NASA and the telemetry industry have led to a special effort to find a solution.

A new high-frequency current standard developed at the Bureau's Radio Standards Engineering Division in Boulder (Colo.) will help satisfy these needs.<sup>1</sup> The new standard permits the measurement of high-frequency current in the range of 1 ampere to 100 amperes at frequencies from 1 MHz to 1 GHz with an uncertainty of less than 1 percent. Developed by N. V. Frederick, it extends still further the Division's capabilities in providing the central core of the national system of electromagnetic measurements.

The new standard is a torque-operated short-circuited-ring electrodynamicometer. The electrodynamicometer consists of a small short-circuited ring situated between the inner and the outer cylinders of a coaxial transmission line, and it measures the current on the transmission line in terms of the torque exerted against the ring by the electromagnetic fields in the line.

Short-circuited-ring electrodynamicometers have, in the past, been proposed as high-frequency current standards because it was thought that the torque-current proportionality constant could be rather easily expressed in terms of the fundamental quantities of mass, length, time, and the permeability of free space. However, some of the basic assumptions which characterized the earlier approaches to this problem of finding the proportionality constant were in error.

In the past, those who have attempted to analyze the short-circuited-ring electrodynamicometer have assumed the torque-current proportionality constant could be determined through an application of Lagrange's equations to the energy expression which describes the free-space transverse electromagnetic-mode fields due to a filament of current near a filamentary ring. The error associated with this approach is that the ring is not a filament; it is also enclosed between coaxial cylinders, and the transverse electromagnetic-mode energy expression does not completely describe the system.

The new method used by Mr. Frederick to obtain the proportionality constant for the short-circuited-ring electrodynamicometer is based on the "resonator action theorem."<sup>2</sup> A. L. Cullen was the first to report an experiment<sup>3</sup> in which this theorem provided a means for obtaining the proportionality constant for an electrical measuring instrument that depended on interactions between the quantity to be measured and a ponderable body. Cullen used the theorem to evaluate the behavior of a torque-operated microwave wattmeter.

The new method of determining the torque-current proportionality constant involves first making the electrodynamicometer into a resonant cavity by fitting movable shorting pistons into the ends of the coaxial transmission line that contains the short-circuited ring. The cavity thus

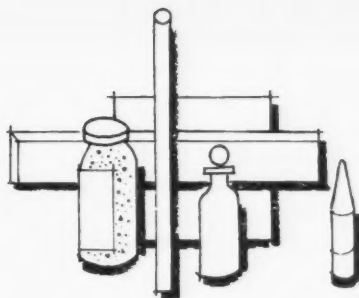
*continued on page 214*

*Sighting through an optical port, N. V. Frederick adjusts a support mechanism in the recently developed high frequency current standard.*





# STANDARD REFERENCE MATERIALS



*Standard Reference Materials are well-characterized materials disseminated by NBS to be used in calibrating and evaluating measuring instruments, methods, and systems or to produce scientific data that can be referred readily to a common base. These materials are certified for chemical composition or for a particular physical or chemical property. They are used on-site in science and industry for calibrating the instruments and methods used for production and quality control of raw materials, chemicals, metals, ceramics, fuels, and radioactive nuclides in manufacturing processes and in research. This column regularly reports on the issuance of new and renewal Standard Reference Materials and on latest developments in the Standard Reference Materials Program.*

The NBS Office of Standard Reference Materials has announced the availability of three new ductile iron standards and a reissued stainless steel standard. These standards are a few of the more than 600 standard reference materials available from the National Bureau of Standards.<sup>1</sup>

## Ductile Iron Standards

The three new ductile iron standards were developed at the request of the Ductile Iron Society and the American Foundrymen's Society. They were designed to meet the urgent needs for reliable instrument calibration, particularly for optical emission and x-ray spectrochemical analysis, in the production control of ductile iron compositions in foundries. These standards, NBS Nos. 1140, 1141, and 1142 (Ductile Iron 1, Ductile Iron 2, and Ductile Iron 3), are certified for carbon, manganese, phosphorus, sulfur, silicon, copper, nickel, chromium, vanadium, molybdenum, titanium, magnesium, and yttrium. The three standards cover the useful concentration ranges for the certified elements. Chemical information is also supplied for aluminum, arsenic, and cerium, although these elements are not now certified.

The material for the standards was melted and cast at the American Cast Iron Pipe Company in Birmingham, Alabama. A special NBS chill-cast mold assembly was used for the preparation procedures similar to those employed for the white cast iron standards.<sup>2</sup>

Results of homogeneity testing at NBS demonstrated that the ductile iron materials are satisfactory for the elements certified. Analyses for certification were performed

at NBS and at the American Cast Iron Pipe Company.

The standards, in the form of chill-cast sections approximately  $1\frac{1}{4}$  inches square and  $\frac{1}{2}$  inch thick, may be purchased for \$60 each.<sup>3</sup>

## Chromium-Nickel Steel Standard

NBS No. 101f is a renewal of stainless steel standard No. 101e, and is an 18Cr-10Ni alloy corresponding in chemical composition to AISI Type 304L. It was prepared primarily for use in calibrating analytical methods used in both production control and customer acceptance of stainless steel.

This renewal, in contrast to previous renewals of this standard, was prepared by a prealloyed powder metallurgical process in which the molten metal was atomized in argon and subsequently annealed in hydrogen. NBS No. 101f is the second ferrous base standard to be prepared by this powder metallurgy process. The first standard to be prepared was NBS No. 163, a 1-percent chromium steel.<sup>4</sup> Extensive investigation of the homogeneity of NBS No. 101f was made in a manner analogous to the homogeneity testing of NBS No. 163. Both NBS and the General Motors Corporation Research Laboratories found the homogeneity to be well within acceptable limits for use as a standard.

NBS No. 101f is in powdered form, sized between 25 and 100 mesh sieves, and is sold in units of approximately 100 grams for \$28 per unit.<sup>3</sup>

Analytical work for certification of NBS No. 101f was performed at NBS and at the GMC Research Laboratories. The standard is being issued initially with a provisional certificate of analysis for carbon, manganese, phosphorus, sulfur, silicon, copper, nickel, chromium, vanadium, molybdenum, and cobalt. For information purposes only, values for tungsten, arsenic, antimony, and gallium have been included, also. A final certificate of analysis for NBS No. 101f is planned for release on or about January 1, 1969.

## Emission Data on Standard Phosphors Issued

The photoexcitation spectral emission properties of 10 of the 14 NBS phosphors have been determined. The data obtained and the techniques used to measure the emission properties under ultraviolet radiation are presented in NBS Technical Note 417, *Spectral Emission Properties* continued

## REFERENCE MATERIAL *continued*

of NBS Standard Phosphor Samples Under Photo-excitation.<sup>5</sup>

Pressed tablets of the phosphors were excited by radiation from a mercury-arc lamp passing through a narrow band-pass filter to obtain either 2537 Å or 3650 Å excitation. The data presented are for the following NBS Standard Reference Materials:

NBS No.	Phosphor	Activator
1020	Zinc sulfide, ZnS	Silver
1021	Zinc silicate, Zn <sub>2</sub> SiO <sub>4</sub>	Manganese
1022	Zinc sulfide, ZnS	Copper
1023	Zinc-cadmium sulfide, ZnCdS	Silver
1024	Zinc-cadmium sulfide, ZnCdS	Copper
1026	Calcium tungstate, CaWO <sub>4</sub>	Lead
1027	Magnesium tungstate, MgWO <sub>4</sub>	None
1028	Zinc silicate, Zn <sub>2</sub> SiO <sub>4</sub>	Manganese

1029	Calcium silicate, CaSiO <sub>3</sub>	Lead, manganese
1031	Calcium halophosphate, 3Ca <sub>3</sub> (PO <sub>4</sub> ) · Ca(F,Cl)	Antimony, manganese

The phosphors are sold for \$18.50<sup>3</sup> per unit without certification to provide persons interested in developing measurement methods for phosphors with a common source of materials. A copy of Technical Note 417 is supplied with each purchase of the phosphor standards.

<sup>1</sup> For a complete list of Standard Reference Materials available from NBS, see Standard Reference Materials: Catalog and Price List of Standard Materials Issued by the National Bureau of Standards, NBS Misc. Publ. 261 (1968 ed.), for sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 45 cents. Quarterly insert sheets which update Misc. Publ. 260 are supplied to users on request.

<sup>2</sup> Standard Reference Materials: Preparation of NBS White Cast Iron Spectrochemical Standards, NBS Misc. Publ. 260-1. Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 30 cents.

<sup>3</sup> These standards may be purchased for the price indicated from the Office of Standard Reference Materials, Rm. B308, Chemistry Bldg., National Bureau of Standards, Washington, D.C. 20234.

<sup>4</sup> See Standard Reference Materials, NBS Tech. News Bull. 52, No. 4, 84-85 (April 1968).

<sup>5</sup> Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, for 25 cents.

## STANDARDS AND CALIBRATIONS *continued*

formed is resonated with the ring in place. The ring is rotated slightly through a small angle,  $\Delta\theta$ , against the torque,  $t$ , which is being exerted against it by the fields within the cavity. This results in work,  $t\Delta\theta$ , being done upon the cavity fields and also results in a change in the total action (period times total energy) of the cavity fields. The pistons are adjusted so as to restore the period to its original value, that is, the value it had before the ring was disturbed. By the resonator action theorem as modified by Cullen, if the cavity losses are sufficiently small, the work done by the pistons being moved a distance,  $\Delta x$ , against the radiation pressure force,  $F$ , must be the negative of the work,  $t\Delta\theta$ , that is,

$$F\Delta x = -t\Delta\theta.$$

Since the radiation pressure against the pistons is easily determined in terms of the current in the cavity, or on the transmission line, the theorem makes possible a convenient means of evaluating the torque acting against the ring in terms of this current.

The new standard will be particularly useful for calibrating instruments that are used to measure high-frequency antenna currents, for measuring the rf currents induced in wires used with electrically detonated explosives, and for calibrating high-frequency current meters. The new standard also serves as a back-up for the NBS high-frequency voltage and power standards. The standard is presently suitable only for primary laboratory application such as at the NBS Radio Standards facility.

## STANDARD FREQUENCY AND TIME BROADCASTS

WWV—2.5, 5.0, 10.0, 15.0, 20.0, and 25.0 MHz

WWVH—2.5, 5.0, 10.0, and 15.0 MHz

WWVB—60 kHz

Radio stations WWV (Fort Collins, Colo.) and WWVH (Maui, Hawaii) broadcast signals that are kept in close agreement with the UT2 scale by making step adjustments of 100 ms as necessary. Each pulse indicates that the earth has rotated approximately 15 arcseconds about its axis since the previous one. The pulses occur at intervals that are longer than one second by 300 parts in  $10^{10}$  due to an offset in carrier frequency coordinated by the Bureau International de l'Heure (BIH), Paris, France. Adjustments are made at 0000 UT on the first day of a month. *There will be no adjustment made on October 1, 1968.*

Radio station WWVB (Fort Collins, Colo.) broadcasts seconds pulses derived from the NBS Time Standard (NBS-III) with no offset. Step adjustments of 200 ms are made at 0000 UT on the first day of a month when necessary. BIH announces when such adjustments should be made in the scale to maintain the seconds pulses within about 100 ms of UT2. *There will be no adjustment made on October 1, 1968.*

<sup>1</sup> Frederick, N. V., A new high frequency standard, IEEE Trans. Instr. Meas. (to be published).

<sup>2</sup> Maclean, W. R., The resonator action theorem, Qrtly. J. Appl. Math. 2, 329-335 (1945).

<sup>3</sup> Cullen, A. L., A general method for the absolute measurement of microwave power, J. IEE (London) 99, Pt. 4, No. 24, 112-120 (1952).

## BRIEFS continued

tion program suitable for use by procurement agencies, by mortgage bankers, and by designers, builders, and owners.

Further program and registration information may be obtained from Robert Cook, Room A600, Administration Building, National Bureau of Standards, Washington, D.C. 20234.

### SCHEDULED NBS-SPONSORED CONFERENCES

Each year NBS sponsors a number of conferences covering a broad range of topics in science and technology. The conferences listed below are either sponsored or cosponsored by NBS and will be held at the Bureau's Gaithersburg, Md., facility unless otherwise indicated. These conferences are open to all interested persons unless specifically noted. For further information, address the person indicated below in care of Special Activities Section, Room A600, Administration Building, National Bureau of Standards, Washington, D.C. 20234.

**Measurements Technology.** Sept. 17-18. Cosponsor: Scientific Apparatus Makers Association. Contact: G. E. Lawrence (SAMA).

**Conference on Thermal Expansion.** Sept. 18-20. Cosponsor: Westinghouse Astronuclear Laboratory. Contact: R. K. Kirby (NBS Metrology Division).

**Performance of Buildings—Concept and Measurement.** Sept. 23-25. Contact: W. W. Walton (NBS Building Research Division).

**1968 International Conference on Modern Trends**

**in Activation Analysis.** Oct. 7-11. Cosponsors: U.S. AEC; International Atomic Energy Agency; EURISOTOP. Contact: P. D. LaFleur (NBS Analytical Chemistry Division).

**Standards for High Pressure Research.** Oct. 14-18. Contact: C. W. Beckett (NBS Heat Division).

**American Cybernetics Association.** Oct. 24-25. Contact: Carl Hammer (UNIVAC).

**Seminar on Durability of Insulating Glass.** Nov. 14-15. Cosponsor: ASTM Committee E-6 on Methods of Testing and Building Construction. Contact: Henry Robinson (NBS Building Research Division).

**Workshop on Mass Spectrometry.** Nov. 18-19. Contact: A. J. Ahearn (NBS Analytical Chemistry Division).

**Symposium on Natural Products.** Dec. 12. Cosponsor: Chemical Society of Washington. Contact: G. Brauer (NBS Polymers Division).

### SPECIAL PUBLICATION SERIES

The NBS Miscellaneous Publication Series has recently been redesignated the NBS Special Publication Series. Because this is merely a title change and not a new series, the numbering sequence will be retained.

The new title is more in keeping with the nature of the material appearing in the series, particularly the proceedings of the national and international meetings the Bureau sponsors. It also provides a more acceptable medium for material not suitable for other NBS publications.

# PUBLICATIONS of the National Bureau of Standards\*

## PERIODICALS

*Technical News Bulletin*, Volume 52, No. 8, August 1968, 15 cents. Annual subscription: Domestic, \$1.50; foreign, \$2.25. Available on a 1-, 2-, or 3-year subscription basis.

*Journal of Research of the National Bureau of Standards*

**Section A. Physics and Chemistry.** Issued six times a year. Annual subscription: Domestic, \$5; foreign, \$6. Single copy, \$1.

**Section B. Mathematical Sciences.** Issued quarterly. Annual subscription: Domestic, \$2.25; foreign, \$2.75. Single copy, 75 cents.

**Section C. Engineering and Instrumentation.** Issued quarterly. Annual subscription: Domestic, \$2.75; foreign, \$3.50. Single copy, 75 cents.

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Ku, H. H., and Kullback, S., Interaction in multidimensional contingency tables: An information theoretical approach.

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[Note: The NBS Miscellaneous Publication Series has recently been redesignated the NBS Special Publication Series. Because this is merely a title change and not a new series, the numbering sequence will be retained.]

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*This column lists all publications by the NBS staff, as soon after issuance as practical. For completeness, earlier references not previously reported may be included from time to time.*

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## CLEARINGHOUSE BIBLIOGRAPHIC JOURNALS\*\*

**U.S. Government Research & Development Reports.** Semi-monthly journal of abstracts of R&D reports on U.S. Government-sponsored projects and U.S. Government-sponsored translations of foreign technical material. Annual subscription (24 issues): Domestic, \$30; foreign, \$37.50. Single copy, \$3.

**U.S. Government Research & Development Reports Index.** Semi-monthly index to preceding; arranged by subject, personal author, corporate author, contract number, and accession/report number. Annual subscription (24 issues): Domestic, \$22; foreign, \$27.50. Single copy, \$3.

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